Impact of Test Dust Changes on Particle Size, Particle Count, and Fluid Cleanliness Classes

Leonard Bensch, Ph.D., PE, and Puliyur Madhavan, Ph.D.
Pall Corporation
25 Harbor Park Drive, Port Washington NY 11050

1998

Abstract: For several decades, AC Fine Test Dust (ACFTD) has been utilized for a number of purposes in the area of hydraulic and lubrication contamination control. It was used for primary calibration of automatic optical particle counters, utilized in laboratories to measure the particle size distribution of a fluid sample, either on-line or collected from the operating system in a sample bottle. ACFTD is no longer being produced and many standards organizations are now selecting replacement dusts, most notably ISO 12103-A3, for calibration and testing purposes. Because none of the replacement dusts has identical particle size distribution characteristics to ACFTD, all associated test results are somewhat different. This paper presents typical changes found with the new dusts including the impact on automatic particle counter calibration, resultant particle sizes, particle counts, and fluid cleanliness classes and codes defined in fluid cleanliness standards commonly used in the industry. It should be pointed out that, although the particle sizes defined by the new calibration, and hence, fluid cleanliness codes or classes, will be changing, this is an artifact of the measurement, and actual contamination levels in the field will remain the same as before.

Key Words: Fluid cleanliness classes; particle counts; particle counter calibration; AC Fine Test Dust; ISO Medium Test Dust

Introduction: Air Cleaner Fine Test Dust, also called AC Fine Test Dust or ACFTD, originally sold by the AC Spark Plug Division (later the AC Rochester Division) of General Motors Corporation, was manufactured by collecting dust, primarily silica, from a certain area in Arizona then ball milling and classifying it into a consistent particle size distribution including particles sizes from roughly 0-100 μm. Because of the consistent particle size distribution of ACFTD and its irregular particle shape, believed to be more representative of contaminants found in typical hydraulic systems, it was chosen in 1969-1970 for the development of a calibration procedure for automatic, liquid borne, optical particle counters, termed APCs. Automatic particle counters are utilized in laboratories and the field to measure the particle size distribution of a fluid sample, either on-line or collected from the operating system in a sample bottle. The calibration procedure, ISO 4402:1991[1], still in use today by most fluid power laboratories around the world, is based on the average longest chord dimension, measured using optical microscopes. The goal of the APC calibration procedure was to ensure that particle counts obtained with an APC agreed as closely as possible with counts obtained by optical microscopy, the most common method employed to obtain particle counts at that time. This particle size

distribution defined in ISO 4402 and shown graphically in Figure 1 is used to set the electronic threshold levels which define the particle sizes measured in a particle counter.

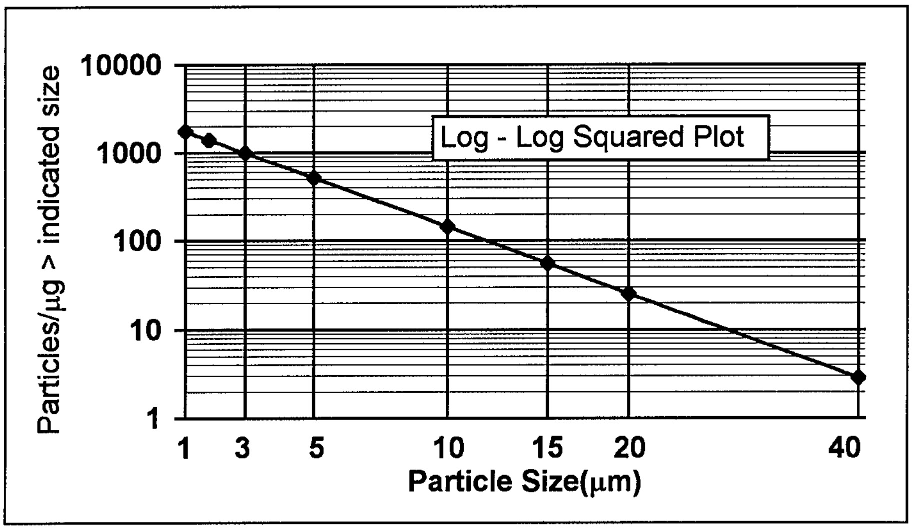

Figure 1. AC Fine Test Dust size distribution based on longest chord dimension.

Since AC Rochester discontinued manufacturing AC Fine Test Dust in 1992, the Society of Automotive Engineers(SAE) in the USA and ISO Technical Committee TC 22 have both made significant efforts to standardize replacement, and additional, test dusts and calibration procedures. Although, at the time of this writing, the modified major test standards have not been officially affirmed, the major portion of the work has been completed. The current status and expected changes are given in the remainder of this paper.

Replacement Test Dusts: The work by SAE and ISO has culminated in the development of a new ISO standard: ISO 12103-1, 1997[2]. This standard defines and designates four new test dusts as listed in Table I.

Table I. Replacement ISO Test Dusts for AC Fine Test Dust.

ISO Designation	Common Name	Other Names
ISO 12103 - A1	ISO Ultrafine Test Dust (ISO UFTD)	PTI 0-10 µm Test Dust
ISO 12103 - A2	ISO Fine Test Dust (ISO FTD)	PTI Fine Test Dust, SAE Fine Test Dust
ISO 12103 - A3	ISO Medium Test Dust (ISO MTD)	PTI 5-80 µm Test Dust, SAE 5-80 µm Test Dust
ISO 12103 - A4	ISO Coarse Test Dust (ISO CTD)	PTI Coarse Test Dust, SAE Coarse Test Dust

The new test dusts are currently being manufactured by Powder Technology Incorporated (PTI) from the same silica based material used by AC Rochester so that the chemical characteristics are similar to the AC Dusts. As opposed to the AC Rochester method, PTI

processes the Arizona dust with a jet mill and classifies it into well-controlled particle size distributions. ISO MTD (ISO 12103 - A3) and ISO FTD (ISO 12103-A2) have the closest particle size distribution to AC Fine Test Dust.

Figure 2 depicts plots of the particle size vs. number distribution of the three dusts in terms of number of particles in one microgram of dust greater than a given particle size. The number of particles in 1 µg of dust is also equal to the number of particles per ml in a 1 mg/L suspension of the dust. The particle size distributions reported in Figure 2 are based on measurements with an automatic particle counter calibrated with ACFTD in accordance with ISO 4402:1991. Neither ISO MTD nor ISO FTD has a particle size vs. number distribution that is equivalent to that of ACFTD. Both ISO MTD and ISO FTD exhibit higher particle counts than ACFTD for sizes below about 10 µm.

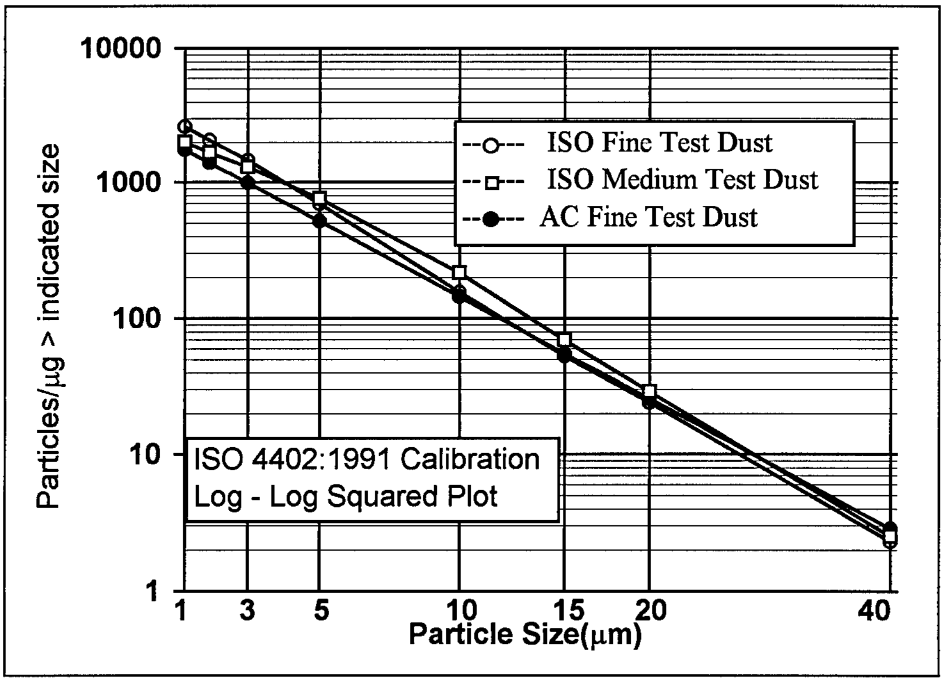

Figure 2. Particle Size *vs* Number Distribution of AC and ISO Test Dusts.

Development of a Revised Calibration Procedure: For some time, the industry had become aware that when more sophisticated instruments such as scanning electron microscopes are used for analysis of ACFTD, a substantial increase in the numbers of particles are reported compared to the distribution based on optical microscopes, given in ISO 4402:1991; this was especially true for particle sizes below about 10 µm. Therefore a project was started at the National Fluid Power Association to develop a new APC calibration method based on a contaminant whose particle size distribution could be traceable to the US National Institute of Standards and Technology (NIST). The result was a new method, ANSI/(NFPA) T2.9.6R1 (1990), that used mono-sized latex particles with sizes traceable to NIST. Usage of this method has been discouraged, since, shortly after its introduction, it was found that poor agreement was obtained between different types of APCs calibrated with latex particles. APCs made by different manufacturers and

APCs utilizing different light sources (such as laser diode or white light) or different measurement principles (light scattering or light extinction) yielded different particle count results when analyzing ACFTD or similar samples. This is due to differences in the optical properties of latex and silica. It was concluded that the APC calibration contaminant should be optically similar to the contaminants typically found in filter testing and field samples.

Based on previous experience, ISO 12103-A3 Test Dust (ISO MTD) was selected as the best candidate due to the fact that it contains less sub-micron particles, which can cause saturation of an automatic particle counter, and is more easily dispersed. A project was undertaken by NIST in 1993 to certify the particle size distribution of suspensions of ISO MTD as a reference material to be used for APC calibration. This effort has resulted in the NIST Standard Reference Material SRM 2806[3] consisting of a 2.8 mg/L suspension of ISO MTD in MIL-H-5606 hydraulic fluid. The results of their analysis show a significant difference in the particle size distribution of ISO MTD as measured with electron microscope (NIST) compared to previous results with an APC calibrated with ACFTD per ISO 4402; see Figure 3. The APC data reported for ISO MTD in Figure 3 are the averages from an international round robin test program sponsored by ISO TC131/SC8/WG9.

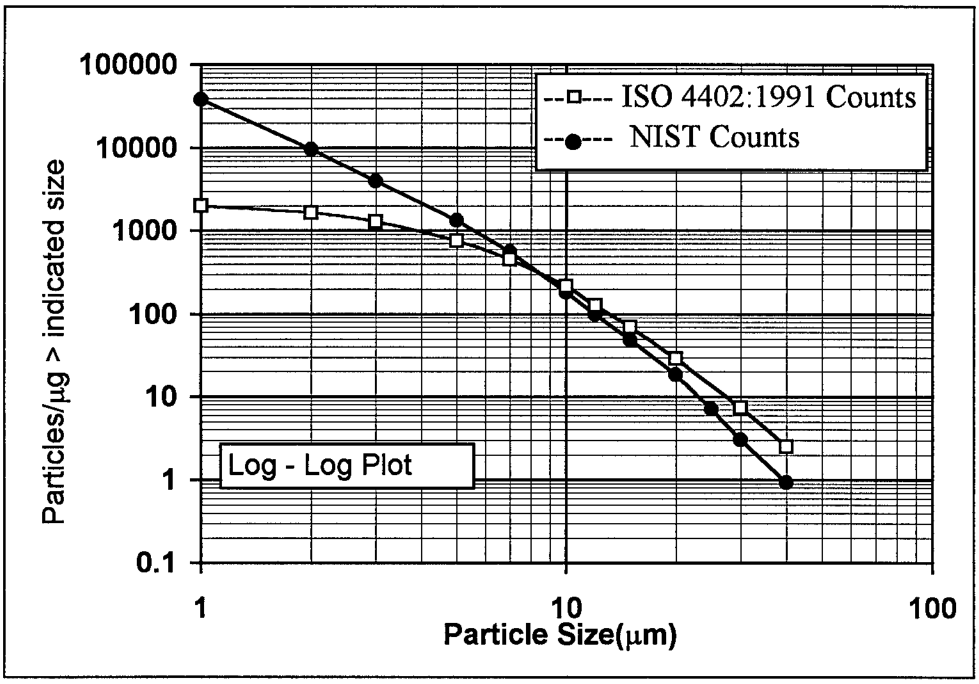

Figure 3. Particle Size *vs* Number Distribution of ISO MTD - ISO 4402 *vs* NIST.

At particle sizes below ~ 10 µm, the NIST size distribution shows significantly higher numbers of particles than the size distribution determined with APCs calibrated per ISO 4402:1991, whereas above a particle size of ~ 10 µm, the NIST distribution shows fewer counts. It should be noted that the NIST measurements were based on the diameter of a sphere whose area is equivalent to the projected two dimensional area of the irregularly

shaped particle; this is the principle of measurement used by most light obscuration automatic particle counters. This differs from the longest chord dimension that ISO 4402:1991 is based upon and is a likely explanation for the discrepancy at particle sizes above ~ 10 μm.

ISO Technical Committee TC131, SC6 has an active project to update the current APC calibration procedure, ISO 4402, using the Standard Reference Material SRM 2806. The revised calibration procedure, circulated for international ballot as DIS 4402:1997[4], includes many other enhancements to ensure better resolution, accuracy, repeatability and reproducibility; however, the effect of the new ISO MTD dust and NIST counts will have the largest impact.

Redefinition of Particle Sizes: Based on the size distributions of ISO MTD in Figure 3, for particle sizes below ~ 10 μm, the particle size determined by NIST for a given particle count (particles/μg) is greater than the corresponding particle size per ISO 4402:1991; the difference increases with decreasing particle size. As an example, the particle count for 1 μm (ISO 4402:1991 calibration) of about 2000 particles/μg corresponds to a particle size of 4.2 μm (NIST size distribution). Thus, below ~ 10 μm, the new definition of the particle size (per NIST size distribution) will be higher than the old definition (ISO 4402), e.g., 2 μm (old) is 4.6 μm (new) and 5 μm (old) is 6.4 μm (new). Above ~ 10 μm, the opposite relationship exists in that new particle sizes will be lower than the old definition, e.g., 15 μm (old) is about 13.6 μm (new). The relationship between particle sizes defined by the ISO 4402:1991 and NIST size distributions for ISO MTD is shown in Figure 4, below, and in Table II.

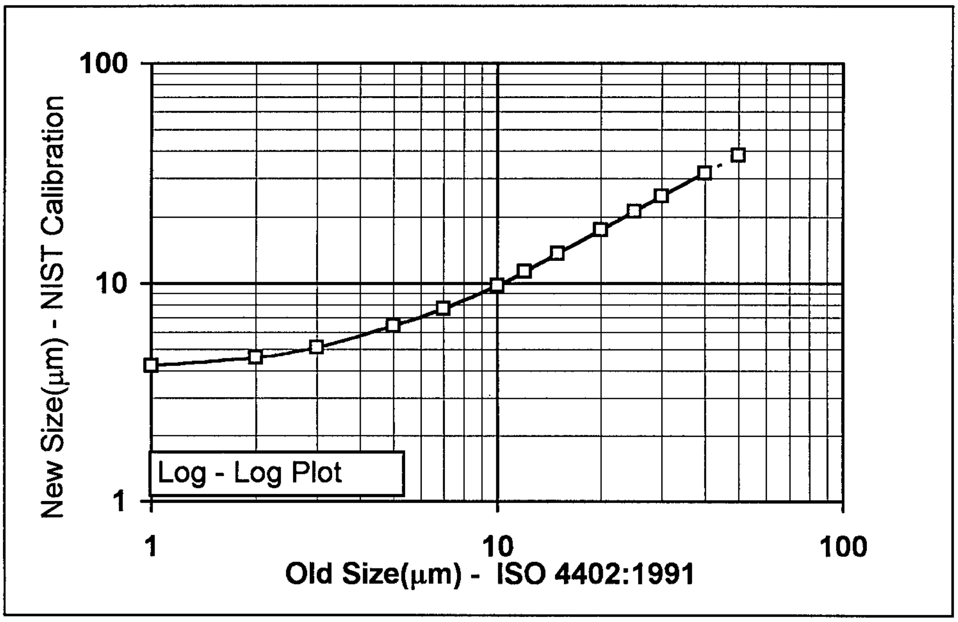

Figure 4. Estimated Changes to Particle Sizes with New Calibration (NIST).

Table II. Comparison of ISO 4402:1991 and NIST Particle Sizes for ISO Medium Test Dust.

ISO 4402:1991 Particle Size(µm):	1	2	3	5	10	15	20	25	40	50
NIST SRM 2806 Particle Size(µm):	4.2	4.6	5.1	6.4	9.8	13.6	17.5	21.2	31.7	38.2*

* Extrapolated value; Figure 4.

Note that because of possible differences between particle counters and the accuracy of their original APC calibration, these relationships may vary slightly and must be determined for each APC to be used. It should also be noted that, even though a particle size or count may vary because of the new calibration, the actual contamination level in a system will not be influenced and will remain the same.

Impact of New Calibration on Particle Counts: The particle counts obtained with an APC calibrated with the new procedure will differ from the corresponding particle counts obtained with the APC calibrated per ISO 4402:1991 at any particular size. Users of particle count data must be made aware of the APC calibration method and how to interpret results when using the new method as compared to ISO 4402:1991. As a first approximation, historical particle count data may be converted from ISO 4402:1991 sizes to the new NIST sizes using Table II.

Figure 5 presents the effect of the two calibration procedures on apparent particle count data. As shown in the figure, if one changes from ISO 4402:1991 to the new NIST calibration without making adjustments to the sizes being monitored, significant

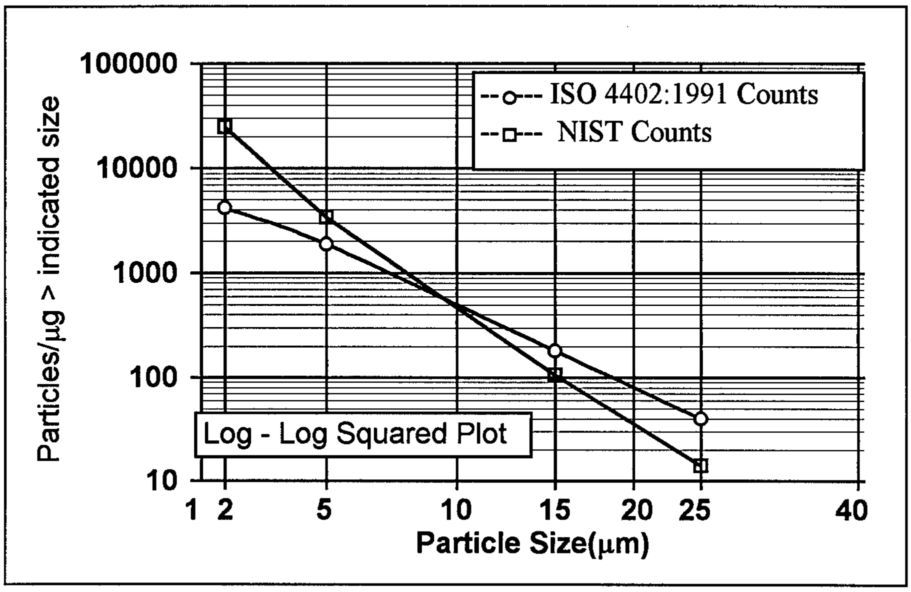

Figure 5. Typical Effect of New Calibration on Particle Counts.

differences arise in particle concentrations. For particle sizes smaller than about 10 μm, apparent increases in particle concentration will be reported which could prompt unnecessary action. The magnitude of the difference increases with decreasing particle size. For sizes larger than 10 μm, the reverse occurs and apparent decreases in concentration result from the new calibration. Failure to recognize this variation as the result of a change in calibration, rather than a change in contamination level, may lead to misinterpretation of particle count data and inappropriate action.

Aerospace Fluid Cleanliness Classification Standards: The NAS 1638 cleanliness standard[5] is comprised of fluid cleanliness classes, each class defined in terms of maximum allowed particle counts for designated particle size ranges. Table III is a partial table of NAS 1638 classes. Cleanliness classes extend from Class 00 to Class 12. For convenience, Classes 3 - 10 have been omitted in the table.

Table III. NAS 1638 Cleanliness Classes.
(Maximum Contamination Limits Based on a 100 mL Sample Size)

Particle Size Range	Classes					Classes	
(μm)	00	0	1	2		11	12
5 to 15	125	250	500	1000		512,000	1,024,000
15 to 25	22	44	89	178		91,200	182,400
25 to 50	4	8	16	32		16,200	32,400
50 to 100	1	2	3	6		2,880	5,760
Over 100	0	0	1	1		512	1,024

The SAE AS4059 and ISO 11218 Standards [6,7] are derived from the NAS 1638 Standard by: 1) Replacing the differential particle size ranges and particle count limits by cumulative particle size ranges and particle count limits, 2) Extending the lower size range to include a > 2 μm size range, 3) Deleting the 50-100 μm size range and replacing the 100+ μm size range by a 50+ μm size range, and 4) Extending the lower limit of the cleanliness classes to include a 000 Class.

Table IV. SAE AS4059 and ISO 11218 Cleanliness Classes.
(Maximum Contamination Limits Based on a 100 mL Sample Size)

Size	Class					Class	
(μm)	000	00	0	1		11	12
> 2	164	328	656	1310		1 340 000	2 690 000
> 5	76	152	304	609		623 000	1 250 000
> 15	14	27	54	109		111 000	222 000
> 25	3	5	10	20		19 600	39 200
> 50	1	1	2	4		3 390	6 780

Table IV is a partial table of AS 4059 and ISO 11218 classes. Cleanliness classes extend from Class 000 to Class 12. For convenience, Classes 2 - 10 have been omitted in the table. Unlike NAS 1638, the cleanliness class of a fluid sample is assigned based on the cumulative particle counts corresponding to a specific cumulative particle size range, with the > 5 μm size range being the default particle size range. Other cumulative particle size ranges in Table IV may be specified as the reference particle size range at the discretion of the user.

Impact of New Calibration: As discussed above, particle counts obtained with an APC calibrated with NIST SRM 2806 would be expected to be significantly higher for particle sizes below ~ 10 μm, and somewhat lower for particle sizes above ~ 10 μm, compared to the particle counts determined with ISO 4402:1991 calibration when the same reference size ranges are used with either calibration. Thus, the cleanliness class of the fluid sample would differ from historical data based on ISO 4402:1991 calibration. Since the actual cleanliness level of the sample is unchanged, it would be necessary to change cleanliness level specifications across the industry to compensate for the change in calibration so that actual cleanliness levels remain unchanged, an approach that is considered impractical from the point of view of implementation. In addition, the 2 μm particle size with the NIST calibration is equivalent to much less than the 1 μm particle size based on ISO 4402 calibration (Table II; Figure 4). Most light obscuration type automatic particle counters would not be able to measure this size, as they are typically limited at about 1 μm based on ISO 4402.

In order to implement the new NIST calibration with minimal impact on cleanliness classes, it is proposed to revise the reference particle size ranges in NAS 1638, AS4059 and ISO 11218, based on the particle size relationship in Table II, such that the new particle size range with the NIST calibration is nearly equivalent to the particle size range with ISO 4402:1991 calibration. As an example, 5 μm per ISO 4402:1991 calibration is replaced with 6 μm (6.4 μm rounded to the nearest integer) per NIST calibration. Because optical microscopic particle counting procedures are not being changed, no changes are required in the cleanliness standards provided the size ranges counted correspond to the original size ranges set by the standards. Table V exemplifies the change

Table V. Modification of SAE AS4059 Cleanliness Classes.

Particle Size(μm)		Class					Class	
Optical Microscope Count	**NIST SRM 2806 Calibration**	**000**	**00**	**0**	**1**		**11**	**12**
> 2	**> 4**	164	328	656	1310		1 340 000	2 690 000
> 5	**> 6**	76	152	304	609		623 000	1 250 000
> 15	**> 14**	14	27	54	109		111 000	222 000
> 25	**> 21**	3	5	10	20		19 600	39 200
> 50	**> 38**	1	1	2	4		3 390	6 780

proposed in the AS4059 cleanliness classes (Table IV) for APC analysis if the approach discussed above is adopted. It should be noted that, since the NIST size distribution does not extend to the 100 μm size range, revision of NAS 1638, as in Table V, would require deletion of the > 100 μm size range and replacement of the 50-100 μm size range with the > 50 μm size range, the same as AS4059 and ISO 11218. The new NIST size ranges for NAS 1638 would then become 6-14 μm, 14-21 μm, 21-38 μm, and > 38 μm.

Industrial Fluid Cleanliness Classification: The ISO Cleanliness Code - ISO 4406[8] is extensively used in the industrial hydraulic and lubrication segment. It is a two number code, e.g., 14/12, based on the number of particles greater than 5 μm and 15 μm respectively. It was expanded to three numbers (ISO DIS 4406, 1994)[9] by the addition of a code number representing the number of particles greater than 2 μm. This standard was approved by international ballot but was subsequently withdrawn prior to publication because of the changes which were imminent to particle size definitions due to the new APC calibration procedure.

The ISO Committee TC131/SC6 has now issued for ballot a modified coding method based on the new calibration procedure. For APC counts, the revised procedure, ISO DIS 4406:1998[10], uses three code numbers, corresponding to the concentrations of particles larger than 4 μm, 6 μm and 14 μm with the new calibration method. The new 6 μm and 14 μm sizes correspond to ISO 4402:1991 sizes of approximately 5 μm and 15 μm (see Table II). These sizes were chosen so that no significant shift in code number occurs due to changes in the APC calibration method. For optical microscopy measurements per SAE ARP598[11] or ISO 4407[12], the calibration is unchanged and the two digits will remain the same as before at 5 μm and 15 μm. Thus the second two digits of the actual code will be similar regardless of the calibration or measurement technique used. The new digit corresponding to 4 μm for APC counts will not be used for microscopic counts.

Conclusion: Replacement contaminant(s) for AC Fine Test Dust, used in the fluid power industry, are necessitated because the dust is no longer being manufactured. In addition, the dust obsolescence as well as a desire to improve APC calibration has resulted in a new calibration procedure that is traceable to NIST. The new NIST calibration procedure will cause numerous changes in the area of contamination control including:

- Particle size definition
- Particle counts
- Cleanliness classes and codes

The projected impact on each of these parameters has been discussed in this paper. Organizations involved in revision of the standards discussed in this paper are aware that there is likely to be a great deal of confusion within the fluid power industry as a result of ACFTD replacement. This is the primary reason that the standards are being revised to minimize changes where possible. Users should always keep in mind that, although laboratory measurements of the particle size distribution in fluid samples (and potentially cleanliness classes) may change with the new calibration, this is an artifact of the

measurement only, and actual contamination levels in the field will remain the same as before.

References

[1] ISO 4402 (1991), Hydraulic fluid power - Calibration of automatic-count instruments for particles suspended in liquids - Method using classified AC Fine Test Dust contaminant.

[2] ISO DIS 12103-1 (1997), Road vehicles - Test dust for filter evaluation - Part 1: Arizona test dust.

[3] National Institute of Standards and Technology - Standard Reference Material® 2806 - Medium Test Dust(MTD) in Hydraulic Fluid, (1997).

[4] ISO DIS 4402 (1997), Hydraulic fluid power – Calibration of liquid automatic particle counters.

[5] NAS 1638 (1992), Cleanliness Requirements of Parts Used in Hydraulics Systems.

[6] SAE AS4059B (1995), Aerospace - Cleanliness Classification for Hydraulic Fluids.

[7] ISO 11218 (1993), Aerospace - Cleanliness classification for hydraulic fluids.

[8] ISO 4406 (1987), Hydraulic fluid power - Fluids - Method for coding level of contamination by solid particles.

[9] ISO DIS 4406 (1994) - Hydraulic fluid power - Fluids - Code for defining the level of contamination of solid particles, Revision of first edition (ISO 4406: 1987).

[10] ISO DIS 4406 (1998), Hydraulic fluid power - Fluids - Code for defining the level of contamination of solid particles.

[11] SAE ARP598B (1986), The Determination of Particulate Contamination of Liquids by the Particle Count Method.

[12] ISO 4407 (1991), Hydraulic fluid power - Fluid contamination - Determination of particulate contamination by the counting method using a microscope.